# Discoloration of PMMA, composite, and PEEK

Heimer, Sina ; Schmidlin, Patrick R ; Stawarczyk, Bogna

Abstract: INTRODUCTION To assess the discoloration and stain removal potential of different cleaning methods relevant to individual/professional prophylaxis and laboratory cleaning on polyetheretherketone (PEEK), poly(methyl methacrylate) (PMMA)-based, and composite (COMP) materials after storage in different media for 7 days. METHODS One thousand three hundred twenty specimens of PEEK, PMMA, and COMP (N = 440 of each group) were prepared and stored in four different media for 7 days to cause stain. Samples were divided into three cleaning groups (n = 10): (i) individual prophylaxis, (ii) laboratory protocols, and (iii) professional prophylaxis. Color was determined by a portable spectrophotometer and calculated between different time points ( E). The data was statistically evaluated using univariate analyses, Kruskal-Wallis H and Mann-Whitney U tests ($p < 0.05$). RESULTS The significantly ($p < 0.001$) lowest discoloration was found when specimens were stored in distilled water and chlorhexidine (CHX), followed by red wine. Curry solution caused the highest discoloration. PEEK showed the significantly ($p < 0.001$) lowest color changes, while COMP showed the highest changes. Ultrasonic bath and Air Flow Plus (AFP) were the significantly ($p < 0.001$) most effective methods to remove staining. The least cleaning effect was found using a soft toothbrush (ST), a medium-hard toothbrush (MT), and SunSparkle (SS) cleaning system. CONCLUSIONS PEEK seems more stable against discolorations than other denture resin materials. Regarding the cleaning potential, individual prophylaxis can be conducted with toothbrushes. For professional prophylaxis, air-abrasion devices using gentle powders are effective. Laboratory protocols should include gentle cleaning methods like ultrasonic bath. CLINICAL RELEVANCE Clinicians and dental technicians should inform their patients about the discoloration potential of certain foods/beverages and recommend the most efficient cleaning, but preventive methods.

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# Discoloration of PMMA, composite and PEEK

Short title: Cleaning methods for PEEK

Sina Heimer[a], Patrick R. Schmidlin[b], Bogna Stawarczyk[a]

[a]Department of Prosthodontics, Ludwig-Maximilians-University, Munich, Germany.
[b]Clinic of Preventive Dentistry, Periodontology and Cariology, Center of Dental Medicine, University of Zurich, Switzerland.

Keywords: cleaning, surface roughness, surface free energy, discoloration, polyetheretherketone

Correspondence

Bogna Stawarczyk, Department of Prosthodontics, Dental School, Ludwig-Maximilians University Munich, Goethestrasse 70, 80336 Munich, Germany, Tel. +49-89-51609573, e-mail: bogna.stawarczyk@med.uni-muenchen.de

**ABSTRACT (257 words)**

***Introduction*** To assess the discoloration and stain removal potential of different cleaning methods relevant for individual/professional prophylaxis and laboratory cleaning on polyetheretherketone (PEEK), PMMA-based (PMMA) and composite (COMP) materials after storage in different media for seven days.

***Methods*** One-thousand-three-hundred-and-twenty specimens of PEEK, PMMA and COMP (N=440 of each group) were prepared and stored in four different media four seven days to cause stain. Afterwards, samples were divided into three cleaning groups (n=10): (i) individual prophylaxis, (ii) laboratory protocols and (iii) professional prophylaxis. Color was measured by a portable spectrophotometer and calculated between different time points ($\Delta E$). The data was statistically evaluated using multivariate analyses, Kruskal–Wallis-H and Mann–Whitney-U tests ($p<0.05$).

***Results*** The significantly ($p<0.001$) lowest discoloration was when specimens were stored in distilled water and CHX, followed by red wine whereas curry solution caused the highest discoloration. PEEK showed the significantly ($p<0.001$) lowest color changes, while COMP showed the highest rate. Ultrasonic bath and AFP were the significantly ($p<0.001$) most effective methods to remove staining. The least stains were removed using a soft toothbrush (ST), a medium-hard toothbrush (MT) and SunSparkle cleaning system (SS).

***Conclusions*** PEEK seems more stable against discolorations than other denture resin materials. Regarding the cleaning potential, individual prophylaxis can be conducted with toothbrushes. For professional prophylaxis, air-abrasion devices using gentle powders seem effective. Laboratory protocols should include gentle cleaning methods like ultrasonic bath.

***Clinical relevance*** Clinicians and dental technicians should inform their patients about the discoloration potential of certain foods/beverages and recommend the most efficient cleaning methods, which have no negative influence on material characteristics.

## 1. Introduction

Polyetheretherketone (PEEK) was originally developed for space travel and introduced to the dental market as a high-performance, chemically inert biomaterial known for its bioinert characteristics [1]. With its extremely low potential to trigger an allergy, PEEK is known to cause very few reported or no systemic immune responses after intraoral insertion [2].

Used in oral rehabilitation, polyetheretherketone (PEEK)-based materials are applied in addition to other polymers like poly(methyl methacrylate) (PMMA)- and composite resin materials upon other terms in RDP and FDP technology. PEEK has become an alternative to conventional and well-investigated veneering and denture base resin materials with low discolorations and improved mechanical properties [3-5]. In order to achieve that, there are various possibilities of compounding processes [6]: in addition to unfilled versions, PEEK is available containing fibers or powder in differing combinations, e.g. carbon or glass fibers in short or endless chain structures reducing wear and improving its mechanical behavior like flexural strength and hardness [7]. High-order restoration materials are characterized by functional aspects like good polishing ability, low water sorption, minimal polymerization shrinkage, low residual monomer content and color stability [8-10]. Not only restoration polishing and material composition but also patient's dietary and cleaning habits are key parameters achieving color stability mainly over a longer period of time [11,12]. Serving as a decisive aesthetic parameter, both patients and dentists expect tooth-colored fillings and natural dental prostheses, which represent important parameters for patient satisfaction and compliance. In order to change the intrinsic color of PEEK restorations, which have a grayish-brown or pearl-white opaque color, several manufacturers of dental materials started to add titanium oxide to achieve a lighter or more tooth-colored appearance [6,13,14]. Another possibility would be veneering with composite resins after different surface modifications [15-17].

Discoloration of dental prostheses developing over a certain period of time is measured as value over time by a spectrophotometer [18| and is determined by two essential factors, i.e. intrinsic and extrinsic influences [5,19]. Extrinsic factors derive primarily from colorants of food products like warm and cold caffeine-containing beverages or mouth rinses, which cause staining by chemical adhesion [20]. But also smoking leads to discolorations on teeth and dental restorations. Intrinsic factors are chemical reactions within the restoration material triggered heredity, age and processing modes of placed restorations [21]. The processes which lead to discoloration of PEEK still remain to be elucidated. Color stability significantly depends on surface properties like surface roughness and surface free energy of dental prostheses [22]. Several studies showed that there is a positive correlation between high surface roughness and discoloration of denture resins, which is explained by the large surface with various predilection sites for agglomerating colorants [23,24|. Combined with bacterial adhesion and transformation of biofilm this cannot only result in development of caries and periodontal disease but also in further discoloration caused by metabolic products of bacteria

[25,26].

Therefore, effective cleaning methods are required. Besides individual and professional prophylaxis measures, laboratory cleaning protocols are available. Two types of cleaning alternatives can be distinguished: Dry cleaning methods like manual instruments (curettes) or corundum blasting or wet cleaning methods containing manual or electric tooth brushing using toothpaste and air-abrasion using powder devices [27-32]. Moreover, it must be noted, that the use of abrasion powders and manual cleaning instruments over a certain period of time results in surface damage of natural teeth and inserted restorations [33,34]. In this context, practicability, time requirements and potential damage to the inserted restoration materials by using different cleaning methods are crucial parameters, which need to be considered carefully. At the present time, however, there are no studies available assessing the cleaning protocols of PEEK and its discoloration or cleaning rate.

This study therefore aimed to assess the discoloration and the stain removal potential of different cleaning methods relevant for individual/professional prophylaxis and laboratory cleaning on polyetheretherketone (PEEK), PMMA-based (PMMA) and composite (COMP) materials after storage in different media for seven days. The null-hypothesis tested was that PEEK shows similar discolorations after storage in different media and after use of various cleaning devices as compared to PMMA- and composite (COMP)- based materials.

## 2. Material and methods

This study examined the influence of eleven different cleaning protocols (3 individual prophylaxis, 4 professional prophylaxis and 4 laboratory protocols) on the surface discoloration of PEEK (bioHPP, bredent, Senden, Germany). The results were compared with two denture resin-based materials, i.e. a cold-curing denture polymethylmethacrylate (PMMA; uni.lign PF 20, bredent) and a veneering resin composite (COMP; crea.lign, bredent) as described in Table 1.

### 2.1 Specimen preparation

Cylindric PEEK blanks were provided by the manufacturer, which were prepared in disc form with a diameter of 15 mm and a thickness of 3 mm (n=440). Corresponding silicone models were used as templates for the PMMA (n=440) and COMP (n=440) specimen fabrication. For the fabrication of the standardized PMMA discs, molds were filled with a PMMA mixture consisting of powder (13 g) and liquid (9 ml) and were polymerized in a pressure pot (palamat elite, Heraeus Kulzer, Hanau, Germany) for 20 minutes at 4.5 bar in 55°C warm water. In order to prepare COMP specimens, the veneering resin composite was filled into the molds with a layer thickness of approximately 1 mm in increments. Each layer was light cured for 180 s at a wavelength of 370 – 500 nm using bre.Lux Power Unit (bredent). After checking that the specimens ranged in the same dimensions using a caliper (+/- 0.05 mm), they were grinded with a series of silicone carbide abrasive papers up to P4000 using a laboratory polishing machine (Abramin, Struers, Ballerup, Danemark). All specimens were polished in the following order: P1200 (3 bar) for 1 min, P4000 (3 bar) for 4 min and P4000 (5 bar) for 4 min with a pressure of 3 bar under constant water-cooling.

### 2.2 Media Storage

The specimens of each group were randomly divided into three subgroups (n=110) consisting of different storage media: (A) red wine (Cepa Lebrel 2013, Lidl GmbH & Co.KG), (B) curry (Ostmann GmbH) and (C) chlorhexidine (Orbi-CHX; Orbis Dental GmbH). Ten control samples of each material (n=10) were stored in distilled water (Aqua Bidest, Kerndl, pH=6.7). While the red wine and chlorhexidine were not diluted, 40 g of the curry powder was suspended in 1 L of distilled water, which was boiled for 10 min. Specimens were then stored at 37 °C for 7 days.

### 2.3 Cleaning methods

<u>*Individual prophylaxis*</u>

Specimens were brushed for 4 minutes with rotary movements and an average contact pressure of 80 g using a toothpaste slurry (blend-a-med complete, procter & gamble GmbH), which was a mixture of a high-abrasive tooth paste

(RDA = 100, 1450 ppm NaF) and tap water (pH value of 7.58) in a ratio of 1:2. The following commercially available toothbrushes were used:

(ST) A soft toothbrush (Dr.Best, GlaxoSmithKline, Munich, Germany) with rounded bristles and a special suspension avoiding on teeth and surrounding gum.

(MT) A medium-hard toothbrush (Dr.Best, GlaxoSmithKline) with same configuration like the soft one different in hardness of bristles.

(SOT) A sonic toothbrush (Oral-B Pulsonic, procter & gamble, Ohio, US) with 31.000 vibrations per minute. The brushes were fixed in special devices (custom-made device at the Ludwig-Maximilians University of Munich), whereby six toothbrushes were serially connected in order to ensure a standardized contact pressure and surface distance.

*Laboratory protocols*

(SY) Sympro (Renfert, Uhldingen-Mühlhofen, Germany): A high-performance cleaning unit for dentures and orthodontic appliances was used with 75 g of needles and 200 ml fluid for 20 minutes at a rotation speed of 2.000 U/min. The specimens were rinsed with cold tap water after cleaning procedure immediately.

(SS) SunSparkle (Sun Dental Laboratories, Düsseldorf, Germany): A ultrasonic battery-operated cleaning unit was tested with tap water and 2.5 g of SunSparkle concentrated disinfecting cleaning powder (Sun Dental Laboratories) for 15 minutes, respectively. For removal of powder residues, specimens were rinsed with cold tap water.

(UB) An ultrasonic bath (USR2200, Dema, Mannheim, Germany) was filled with tap water and specimens were cleaned for 380 seconds according to the manufacturer`s instructions. Technical data: power, 170 W; cleaning frequency, 42 kHz; capacity, 2600 ml.

(AP) Aluminum oxide blasting (50 µm) (Renfert): Using the Basic quattro IS (Renfert) specimens were cleaned for 15 seconds with aluminum oxide (50 µm) at a distance of 4 mm with following operating parameters: Working pressure, 14.5-87 psi; connecting pressure 87-116 psi. Aluminum oxide particles (Cobra white, Renfert) were applied in sharp-edged and extremely pure form (approx. 99.7% $Al_2O_3$).

*Professional prophylaxis*

(PS) Perio Soft-Scaler (Kerr, Karlsruhe, Germany): Specimens were cleaned for 15 seconds applying a reaming motion with an average contact pressure of 80 g.

(SO) Sonicflex 2003/L (KaVo Sonicflex, Biberach, Germany): An Air Scaler with a cellular optic glass rod (SONICflex 2003L, KaVo) and the Sonicflex tip #7 was used for 15 seconds with rotary movements at sound frequency. Operating parameters: operating pressure, 36 psi; performance level 1; the average contact pressure was 80 g and the water setting at 15 psi.

(AFC) Air Flow Comfort (EMS, Nyon, Switzerland): The powder was applied in a PROPHYflex 3 (KaVo) with a handle sleeve, which can be rotated by 360 degrees. The construction of the cannula guaranteed a concentrated powder jet. The following operating parameters were used: drive pressure, 46-73 psi; air consumption, 10-13 Nl/min; nozzle angle, 60° and the nozzle-surface distance, 4mm. The supragingival sodium bicarbonate air polishing powder (40 µm) was used for 15 seconds in moving circles.

(AFP) Air Flow Plus (EMS): The powder was applied in a PROPHYflex 3 (KaVo) too. The powder, which is suitable for supra- and subgingival polishing (14 µm) was tested in analogy to AFC.

For reducing the outcome variability to a minimum, all preparations, cleaning methods and evaluations were performed by the same person (SH).

### 2.4 Color measurements

The color and color difference measurements were performed by a portable spectrophotometer SP60 (X-rite GmbH, Michigan, USA) at three different time points, i.e. before and after storage in different media and after final cleaning. The following operating parameters were used: Standard illuminant D65, illuminating geometry d/8° and standard observer 10°. Reflectance and transmission measurements were combined formulating translucency. After calibration of the colorimeter according to the manufacturer´s recommendations, the target window was directly applied on the specimen surface at three different varying areas and measurements were started under daylight conditions on the same time of three consecutive days. Light source and samples were at a 90°angle to each other.

A formulation Master FM 2 software (X-rite GmbH) was used generating three separate values containing L* (degree of lightness), a* (degree of greenness or redness) and b* values (degree of blueness or yellowness) according to the CIELAB color system (Commission Internationale de l´Eclairage, CIE). The individual values L*, a* and b* are not sufficient enough to indicate the color changes. For this reason ΔE* values were generated, which represent the differences between the upper-mentioned values: ΔE1 (before and after media storage), ΔE2 (before and after cleaning procedures) and ΔE3 (before media storage and after cleaning). The discoloration (ΔE*) of each specimen was calculated using the following formula [36]:

$$\Delta E^*=\sqrt{(\Delta L^*)^2+(\Delta a^*)^2+(\Delta b^*)^2}$$

*Thereby, differences in coloration on the basis of media storage and their changes by cleaning could be determined and compared.*

## 2.5 Statistical methods

The effect of the independent parameters of storage medium, cleaning protocol and material group as well as the effect of their interaction on discoloration was assessed using multivariate analyses. Normality of data distribution was tested using the Kolmogorov–Smirnov test. Kruskal–Wallis-H and Mann–Whitney-U tests were used for analyze the effect of the storage media, cleaning protocols and materials ($p<0.05$). Statistical analyses were performed using software SPSS Version 23.0 (SPSSINC, Chicago, IL, USA).

## 3. Results

### 3.1 Discoloration after 7 days storage in different media (ΔE1) (Fig. 1)

Data regarding the discoloration after 7 days storage in different media is presented in Figure 1.

The storage medium and polymer material showed a significant ($p<0.001$) impact on the discoloration values. However, storage media exerted higher partial eta squared values ($\eta P^2=0.599$) than the polymer materials ($\eta P^2=0.036$).

The significantly ($p<0.001$) lowest discolorations were observed for specimens, which were stored in distilled water and chlorhexidine, followed by red wine. The significantly ($p<0.001$) highest one was observed after storage in curry solution. The discoloration by specimens stored in distilled water was so low that it was not assessed in further measurements after cleaning procedures.

In general, PEEK showed significantly ($p<0.001$) lower discolorations as compared to PMMA and COMP. COMP showed the highest discoloration potential.

### 3.2 Discoloration after different cleaning procedures (ΔE2) (Table 2)

Data regarding the discoloration after different cleaning procedures is presented in Figure 2.

Storage medium, polymer material and cleaning procedure showed a significant ($p<0.001$) impact on the discoloration values after cleaning procedure. The storage medium exerted the highest influence on the discoloration ($\eta P^2=0.519$), closely followed by the cleaning procedure ($\eta P^2=0.151$) and the polymer material ($\eta P^2=0.020$).

Storage in chlorhexidine led to a significantly ($p<0.001$) lower discoloration than storage in red wine. Specimens stored in curry solution showed again the highest discoloration. Ultrasonic bath and Air Flow Plus were the significantly ($p<0.001$) more effective methods to remove extrinsic staining. The weakest discoloration removal potential was shown for the soft and medium-hard toothbrushes as well as for Sympro ($p<0.001$). After cleaning, PEEK and PMMA showed significantly ($p=0.008$) lower discoloration as compared to COMP.

### 3.3 Discoloration differences between before and after storage and cleaning procedure (ΔE3) (Table 3)

Data showing the results of the discoloration differences before and after storage and the different cleaning procedures is presented in Table 3.

Storage medium, polymer material and cleaning procedure showed a significant ($p<0.001$) impact on the ΔE values. The storage medium exerted the highest influence on the discoloration ($\eta P^2=0.381$), closely followed by the polymer material ($\eta P^2=0.375$) and the cleaning procedure ($\eta P^2=0.099$).

Specimens stored in chlorhexidine showed significantly ($p<0.001$) lower ΔE values than specimens stored in red wine or curry solution. No significant ($p=0.064$) differences were observed between specimens stored in two latter solutions.

Specimens cleaned using Sympro, Air Flow Plus, SunSparkle, Air Flow Comfort, as well as all three toothbrush types showed significantly ($p<0.001$) lower $\Delta E$ values than specimens cleaned with ultra sonic bath, Perio Soft-Scaler and $Al_2O_3$ powder. PEEK showed significantly ($p<0.001$) lower $\Delta E$ values than COMP and PMMA. No differences ($p=0.980$) were observed between COMP and PMMA.

## 4. Discussion

### 4.1 Determination of discoloration

The overall appearance and perception of dental restorations depends on several factors: The color of the neighbor teeth or the remaining tooth substance, light scattering effects and inherent material characteristics like opacity and translucency. Detectable color differences to the human eye are normally non discernible below ΔE values of 1, which change into an unacceptable color set at ΔE when more than 3.3 [36-38].

Color change measurements are mainly performed by instrumental or visual methods. Since a subjective visual appraisal is rather difficult for routine application and quantification, instrumental devices are the preferred method for objective evaluation, especially for research purposes [39].

### 4.2 Impact of storage media

In the present study, specimens were stored in media, which are already known to cause discolorations. Especially foods/drinks including pigments like curry and red wine cause accentuated staining by anionic polyphenols in interaction with cationic salivary pellicles [40,41]. In this study, however, specimens were only stored in different media without influence of natural or artificial salivary. Therefore, any discolorations resulted from interactions of food or drink ingredients and the respective material characteristics.

There are three main factors causing extrinsic stain by supporting adhesion of colorant particles on restoration surfaces [42]: Physical and chemical forces like electrostatic forces (van der Waals) and hydrophobic interdependencies. Media with low pH values in the range of 3-6 lead to surface softening and removal of inorganic substances like $Ca^{2+}$, $Al^{3+}$ and $Sr^{2+}$ combined with an increased dye deposition [43,44]. Red wine is more acidic than distilled water or chlorhexidine with a pH of 5.5-7. Despite of alkaline pH values, the curry suspension led to significantly higher color changes than red wine, followed by chlorhexidine and distilled water. This can be explained by the fact that curry includes more orange pigments in contrast to the other media used. Conjugated diarylhepnoids (e.g. curcumin) cause orange color staining and show a high affinity to the polymer phase [45].

Furthermore, physiomechanical characteristics of the tested materials like water absorption, solubility and Martens hardness (HM) must also be considered. Another studies observed no correlations between these properties and the tendency to discoloration [46,47]. PEEK showed the lowest water absorption, solubility and HM values as compared to PMMA-based and composite resin materials. During a storage period of 180 days in saliva and distilled water, water absorption was independent of the storage medium, but showed dependency on storage duration and material properties [48]. The results can be explained by the fact that a higher amount of resin matrix in combination with a lower content of filler particles results in a higher water absorption [49]. These materials are vulnerable to staining by hydrophilic colorants in aqueous solutions increasing over a longer period of storage time [50]. According to another study, water storage and thermal cycling had no influence on HM of tested composite resin materials at all [51].

**4.3 Impact of tested cleaning devices**

The question, however, still remains, how color stain caused by different media can be effectively removed from PEEK restorations once they occur. First of all, a differentiation between three main user groups must be made: Patients, who perform individual prophylaxis using manual or electric toothbrushes at home; dentists, who apply professional prophylaxis measures in their dental practice; and dental technicians, who use laboratory protocols in their laboratory. With regard to the discoloration, only some cleaning protocols showed an effect. Sonic toothbrush, Sympro and Air Flow Plus reflected the most widespread cleaning devices with respect to the individual user group related to stain removal capacity of PEEK. According to a laboratory study assessing extrinsic stain removal, a clear superiority of sonic toothbrushes could be demonstrated so far [52]. Particularly noticeable were the contradictory ΔE2 and ΔE3 values of tested PMMA after cleaning with aluminum oxide (AP). PMMA consists primarily of matrix components with a low filler degree. They are known for their vulnerability for color changes caused by certain beverages [53]. Red wine as a representative of a more acidic medium is able to attack the material`s surface. When aluminum oxide particles impact a material`s surface, a high amount of superficially situated red wine pigments can be removed, which is a possible explanation for the high ΔE2 value. During aluminum oxide impact, energy is liberated in form of heat. This process may also induce changes in the matrix opacity. The result is a strong deviation from the initially measured color, especially in combination with residual and embedded red wine pigments. Therefore, high ΔE3 value can be observed.

Concerning the L, a and b values (CIELAB color system) of the tested materials, a wide variance could be noticed. PEEK (mean L value 88.2) is darker than COMP (mean L value 66.3) and PMMA (mean L value 55.4). Therefore, it must be taken into consideration that brighter materials potentially show a higher tendency for discolorations than darker ones. Therefore, the conditions at baseline for the different materials are not the same and there is some potential to distort the results.

Furthermore, the high deviation of ΔE1 values in the group COMP (figure 2) is striking. COMP shows an average filler content of approximately 40%. Color particles are located between the connected surfaces of filler and matrix. The irregularity of distribution of inserted fillers and pigments correlates with a wider dispersion of the values.

An ongoing in-house study also evaluated the surface roughness and free energy after different cleaning protocols. The above-mentioned cleaning methods resulted in severe surface changes like scratches and notches. Cleaning using conventional air-abrasion and powders (AP), followed by AFC resulted in higher SR values, i.e. with higher values than the recommended threshold value of 0.2 µm, than the other devices (< 0.2 µm) [54]. The latter threshold value is defined as a more or less arbitrarily derived value, from which a significant higher biofilm formation is notable [55]. As a consequence, the use of these cleaning methods should theoretically be avoided. With regard to professional prophylaxis, air-abrasion using sodium bicarbonate powder of specific size is known to cause substance damage on natural teeth. However, it displays outstanding abilities to remove extrinsic stains [56]. The overall degree of substance loss depends on particle size, instrumentation time and working distance [57].

High surface roughness correlates with increased initial biofilm adhesion. It has to be investigated, which parameters predominantly influence bacterial adherence and growth. Previous studies corroborated that PEEK surfaces with a low surface roughness (< 0.2 µm) and free energy showed significantly less bacterial growth [58]. Both, unspecific plaque accumulation and the color transformation process of biofilms cause discoloration. However, the user has to strike a balance between damage and benefit of used cleaning devices.

## 5. Conclusion

Within the limitations of this study, it can be concluded that PEEK showed the significantly ($p<0.001$) lowest discoloration after a one-week storage over all coloring media as compared to PMMA and COMP. PEEK starts with higher L values and a lower tendency to discoloration than COMP and PMMA.

In order to reduce discolorations of PEEK restorations, patients should use sonic toothbrushes within the limitations of application like erosions for individual prophylaxis. Dentists are able to provide color stability of PEEK by utilizing powder air-abrasion e.g. AFP within the scope of professional prophylaxis. For laboratory protocols, the use of Sympro or an ultrasonic bath is recommended.

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## COMPLIANCE WITH ETHICAL STANDARDS

All procedures performed in studies involving human participants were in accordance with the ethical standards of the institutional and/or national research committee and with the 1964 Helsinki Declaration and its later amendments or comparable ethical standards.

## CONFLICT OF INTEREST

All authors declare that they have no conflict of interest.


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**ETHICAL APPROVAL**

This article does not contain any studies with human participants or animals performed by any of the authors.

**INFORMED CONSENT**

For this type of study, formal consent is not required.

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**Table 1 – Used products, manufacturer and compositions.**

| **Material** | **Abbrev.** | **Lot #** | **Manufacturer** | **Composition** |
|---|---|---|---|---|
| bioHPP | PEEK | 410240 | bredent, Senden, Germany | Ceramic filled (20%) PEEK |
| Uni.lign PF 20 | PMMA | 396617/401822 | bredent, Senden, Germany | 99% PMMA polymer |
| crea.lign Incisal E2 | COMP | N141331/123765 | bredent, Senden, Germany | Bis-GMA composite with microfillers |

**Table 2 - Overview of mean values and standard deviations of ΔE2 after different cleaning procedures divided into the different materials (PEEK, PMMA, COMP) and storage media (red wine, curry, CHX). Values about ΔE2 3.3 are written in bold.**

| | | ΔE2 Mean ±SD | | |
|---|---|---|---|---|
| | cleaning method | red wine | curry | CHX |
| **PEEK** | | | | |
| individual PPx | ST | 2.21 ±0.42$^{bB1}$ | **3.61 ±0.45$^{aC6}$** | 0.70 ±0.16$^{bA1}$ |
| | MT | 1.99 ±0.78$^{aB1}$ | **3.30 ±0.34$^{aC56}$** | 0.47 ±0.11$^{aA1}$ |
| | SOT | 2.13 ±0.56$^{bB1}$ | 2.51 ±0.47*$^{abB345}$ | 0.45 ±0.20$^{aA1}$ |
| laboratory protocols | SY | 2.23 ±0.44$^{aB1}$ | 2.69 ±0.34$^{aC456}$ | 0.34 ±0.33*$^{aA1}$ |
| | SS | 1.89 ±0.44$^{aB1}$ | 1.62 ±0.28$^{aB123}$ | 0.17 ±0.11$^{aA1}$ |
| | UB | 2.00 ±0.33$^{cC1}$ | 1.29 ±0.26$^{aB12}$ | 0.28 ±0.22$^{aA1}$ |
| | AP | 2.18 ±0.73$^{aC1}$ | 1.11 ±0.38$^{aB1}$ | 0.35 ±0.48*$^{aA1}$ |
| professional PPx | PS | 2.15 ±0.73$^{bB1}$ | 1.98 ±0.47$^{abB1234}$ | 0.40 ±0.27$^{aA1}$ |
| | SO | 1.64 ±0.73$^{aB1}$ | 1.63 ±0.73$^{aB123}$ | 0.37 ±0.38*$^{aA1}$ |
| | AFC | 1.24 ±0.71$^{aB1}$ | 2.15 ±0.81$^{aC234}$ | 0.37 ±0.26$^{aA1}$ |
| | AFP | 1.18 ±0.65$^{aB1}$ | 2.04 ±0.51$^{aC1234}$ | 0.35 ±0.22$^{aA1}$ |
| **PMMA** | | | | |
| individual PPx | ST | 2.22 ±0.72$^{bB12}$ | 2.49 ±0.38$^{aB45}$ | 0.69 ±0.19$^{bA12}$ |
| | MT | 2.46 ±1.33$^{aB23}$ | 2.58 ±0.34$^{aB5}$ | 1.28 ±0.49$^{bA12}$ |
| | SOT | 1.97 ±0.61$^{abB12}$ | 1.54 ±0.24$^{aB12}$ | 0.66 ±0.39$^{aA1}$ |
| laboratory protocols | SY | 2.14 ±0.82$^{aB2}$ | 2.28 ±0.42$^{aB345}$ | 0.84 ±0.38$^{bA12}$ |
| | SS | 1.90 ±0.35*$^{aB12}$ | 1.53 ±0.45$^{aB12}$ | 0.71 ±0.31$^{cA12}$ |
| | UB | 1.49 ±0.40$^{bA12}$ | 1.26 ±0.33$^{aA1}$ | 1.58 ±1.08$^{bA2}$ |
| | AP | **3.58 ±0.77$^{bC3}$** | 1.72 ±0.45$^{aB123}$ | 0.62 ±0.33$^{aA1}$ |
| professional PPx | PS | 1.08 ±0.35$^{aA1}$ | 1.35 ±0.29$^{aA12}$ | 1.11 ±0.40$^{bA12}$ |
| | SO | 1.53 ±0.52$^{aB12}$ | 1.79 ±0.28$^{aB123}$ | 0.77 ±0.17$^{bA12}$ |
| | AFC | 1.46 ±0.55$^{aB12}$ | 1.84 ±0.24$^{aB1234}$ | 0.56 ±0.27$^{aA1}$ |
| | AFP | 1.35 ±0.50$^{aB12}$ | 1.97 ±0.37$^{aC2345}$ | 0.64 ±0.25$^{bA1}$ |
| **COMP** | | | | |
| individual PPx | ST | 1.42 ±0.51$^{aA123}$ | **5.89 ±2.39$^{bB2}$** | 0.36 ±0.13$^{aA1}$ |
| | MT | 1.60 ±0.24$^{aA123}$ | **5.44 ±3.16$^{bB12}$** | 0.34 ±0.17$^{aA1}$ |
| | SOT | 1.46 ±0.52$^{aA123}$ | **3.70 ±2.03$^{bB12}$** | 0.44 ±0.23$^{aA1}$ |
| laboratory protocols | SY | 2.12 ±0.37$^{aB3}$ | 3.01 ±1.63$^{aB12}$ | 0.43 ±0.32*$^{aA1}$ |
| | SS | 1.84 ±0.34$^{aB23}$ | 1.77 ±0.65$^{aB1}$ | 0.44 ±0.18$^{bA1}$ |
| | UB | 0.86 ±0.40$^{aA1}$ | 2.05 ±1.29$^{aB12}$ | 0.33 ±0.18$^{aA1}$ |
| | AP | 1.94 ±0.89$^{aAB23}$ | 3.08 ±1.93*$^{bB12}$ | 0.53 ±0.38*$^{aA1}$ |
| professional PPx | PS | 0.77 ±0.40$^{aA1}$ | 3.23 ±2.72$^{bB12}$ | 0.67 ±0.37$^{aA1}$ |
| | SO | 1.37 ±0.38$^{aA123}$ | 3.26 ±1.85$^{bB12}$ | 0.43 ±0.15$^{aA1}$ |

| | | | | |
|---|---|---|---|---|
| | AFC | 1.29 ±0.43$^{aA123}$ | 3.16 ±2.03$^{aB12}$ | 0.33 ±0.20$^{aA1}$ |
| | AFP | 1.13 ±0.43*$^{aB12}$ | 2.47 ±0.91$^{aC12}$ | 0.25 ±0.09$^{aA1}$ |

$^{abcd}$ Different superscript lowercase letters represent significant differences between media and cleaning protocols within one material group.
$^{ABC}$ Different superscript uppercase letters represent significant differences between materials and cleaning protocols within one medium.
$^{1234567}$ Different superscript uppercase letters represent significant differences between materials and media within one cleaning protocol.
* not normally distributed data.

**Table 3 - Overview of mean values and standard deviations of ΔE3 after different cleaning procedures divided into the different materials (PEEK, PMMA, COMP) and storage media (red wine, curry, CHX). Values about ΔE2 3.3 are written in bold.**

| | | ΔE3<br>Mean ± SD | | |
|---|---|---|---|---|
| | cleaning method | red wine | curry | CHX |
| **PEEK** | | | | |
| individual PPx | ST | 0.55 ±0.13$^{aA12}$ | 0.38 ±0.09$^{aA12}$ | 0.67 ±0.21$^{aA1}$ |
| | MT | 0.60 ±0.14$^{aB2}$ | 1.20 ±0.28$^{aA1}$ | 0.45 ±0.11$^{aA1}$ |
| | SOT | 0.31 ±0.10$^{aA12}$ | 0.31 ±0.09$^{aB34}$ | 0.34 ±0.17*$^{aA1}$ |
| laboratory protocols | SY | 0.21 ±0.12$^{aA1}$ | 1.70 ±0.37$^{aA1}$ | 0.26 ±0.26$^{aA1}$ |
| | SS | 0.44 ±0.28$^{aA12}$ | 2.04 ±0.41$^{aB45}$ | 0.24 ±0.12$^{aA1}$ |
| | UB | 0.40 ±0.13$^{aA12}$ | 1.67 ±0.35$^{abB5}$ | 0.28 ±0.18$^{aA1}$ |
| | AP | 1.19 ±0.23$^{aB3}$ | 1.37 ±0.19$^{aC45}$ | 0.34 ±0.42*$^{aA1}$ |
| professional PPx | PS | 0.30 ±0.23*$^{aA12}$ | 1.38 ±0.20$^{aB34}$ | 0.39 ±0.29*$^{aA1}$ |
| | SO | 0.30 ±0.10$^{aA12}$ | 1.05 ±0.48$^{aB34}$ | 0.24 ±0.17$^{aA1}$ |
| | AFC | 0.32 ±0.16$^{aA12}$ | 1.40 ±0.29*$^{aB23}$ | 0.39 ±0.22$^{aA1}$ |
| | AFP | 0.25 ±0.19$^{aA1}$ | 0.67 ±0.21$^{aB34}$ | 0.27 ±0.23$^{aA1}$ |
| **PMMA** | | | | |
| individual PPx | ST | 1.48 ±0.39$^{aB12}$ | 1.11 ±0.27$^{aA1}$ | 0.91 ±0.25$^{bA1}$ |
| | MT | 2.36 ±1.03*$^{bB2}$ | 1.63 ±0.31$^{aA12}$ | 1.43 ±0.60$^{bA1}$ |
| | SOT | 1.66 ±0.61$^{bB12}$ | 1.55 ±0.37$^{aB1234}$ | 0.81 ±0.29*$^{bA1}$ |
| laboratory protocols | SY | 1.13 ±0.45$^{abA1}$ | 1.83 ±0.29$^{bA123}$ | 1.14 ±0.33$^{bA1}$ |
| | SS | 0.85 ±1.31$^{aA1}$ | 2.22 ±0.33$^{aB3456}$ | 0.94 ±0.19$^{cA1}$ |
| | UB | 1.41 ±0.22$^{abA12}$ | 2.51 ±0.45*$^{bB4567}$ | 1.57 ±0.79$^{bA1}$ |
| | AP | **4.49 ±0.63$^{bC3}$** | 2.31 ±0.22$^{baB7}$ | 1.03 ±0.56$^{bA1}$ |
| professional PPx | PS | 2.22 ±0.47$^{bB2}$ | 2.42 ±0.40$^{aB567}$ | 1.16 ±0.46$^{bA1}$ |
| | SO | 1.76 ±0.38$^{bB12}$ | 1.70 ±0.27$^{bC67}$ | 0.82 ±0.22$^{cA1}$ |
| | AFC | 1.69 ±0.36$^{bB12}$ | 1.58 ±0.35*$^{bB2345}$ | 0.88 ±0.40$^{bA1}$ |
| | AFP | 1.48 ±0.20$^{bB12}$ | 0.91 ±0.25$^{aB1234}$ | 0.87 ±0.32$^{bA1}$ |
| **COMP** | | | | |
| individual PPx | ST | 2.96 ±1.92$^{bB1}$ | 2.12 ±1.09$^{bAB1}$ | 0.53 ±0.14$^{aA1}$ |
| | MT | 2.02 ±1.09*$^{bB1}$ | 2.41 ±0.92$^{bB1}$ | 0.55 ±0.05$^{aA1}$ |
| | SOT | 2.37 ±0.95$^{bB1}$ | 2.12 ±0.98$^{bB1}$ | 0.63 ±0.22$^{bA1}$ |
| laboratory protocols | SY | 1.74 ±1.56*$^{bAB1}$ | 1.64 ±0.58$^{bB1}$ | 0.52 ±0.19$^{aA1}$ |
| | SS | 2.04 ±1.31$^{bB1}$ | 1.63 ±0.57$^{aB1}$ | 0.56 ±0.15$^{bA1}$ |
| | UB | 2.52 ±1.98*$^{bB1}$ | 1.48 ±0.75$^{aAB1}$ | 0.64 ±0.16*$^{aA1}$ |
| | AP | 1.54 ±0.48$^{aB1}$ | 2.39 ±1.74$^{aB1}$ | 0.68 ±0.26$^{abA1}$ |
| professional PPx | PS | 2.89 ±0.45$^{cB1}$ | 2.14 ±0.84*$^{aB1}$ | 0.61 ±0.47*$^{aA1}$ |
| | SO | 1.95 ±0.46*$^{bB1}$ | 1.52 ±0.64*$^{bB1}$ | 0.56 ±0.16$^{bA1}$ |

| | | | | |
|---|---|---|---|---|
| | AFC | $2.33 \pm 0.65^{*cC1}$ | $1.37 \pm 1.01^{abB1}$ | $0.48 \pm 0.12^{*aA1}$ |
| | AFP | $2.34 \pm 0.43^{cC1}$ | $0.53 \pm 0.14^{*aB1}$ | $0.44 \pm 0.14^{aA1}$ |

$^{abcd}$ Different superscript lowercase letters represent significant differences between media and cleaning protocols within one material group.
$^{ABC}$ Different superscript uppercase letters represent significant differences between materials and cleaning protocols within one medium.
$^{1234567}$ Different superscript uppercase letters represent significant differences between materials and media within one cleaning protocol.
* not normally distributed data.

**Figures and Tables**

**Table 1 –** Used products, manufacturer and compositions.

**Table 2 -** Overview of mean values and standard deviations of ΔE2 after different cleaning procedures divided into the different materials (PEEK, PMMA, COMP) and storage media (red wine, curry, CHX). Values about ΔE2 3.3 are written in bold.

**Table 3 -** Overview of mean values and standard deviations of ΔE3 after different cleaning procedures divided into the different materials (PEEK, PMMA, COMP) and storage media (red wine, curry, CHX). Values about ΔE2 3.3 are written in bold.

**Figure 1.** Study design illustrating the different steps of specimen preparation, media storage, cleaning methods and color measurements.

**Figure 2.** Discoloration rate after 7 days storage in different media (ΔE1).